

TiO₂ Nanotubes Formation by Two-Step Anodization

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Keywords: TiO₂; anodization

ABSTRACT – TiO₂ nanotubes were synthesized by two-steps anodization on Ti foil. Two-steps anodization process focuses on the growth of titania nanotubes through the aid of concaves left by the removal of nanotubes produced via the first step anodization. The growth of TiO₂ length and diameter produced by this anodization is expected to be directly proportional to the anodizing time and voltage. The morphological and structural properties of the nanotubes produced were observed and studied by using field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and Raman spectroscopy technique.

1. INTRODUCTION

One-step anodization (OSA) and two-step anodization (TSA) have been widely used to produce anodic TiO₂ nanotubes. Utilizing one-step anodization, the fabricated TiO₂ NTs commonly demonstrate distinction in tube lengths and nearly irregular surfaces. It has been currently proved that a two-step anodization is feasible for formation of highly ordered and uniform TiO₂ NTs [1]. Due to the systematic distribution across the surface of the pre-treated Titanium foil at the early start, the regularity and orderliness of the nanotube arrays were established during the two-step anodization [2]. It has been delineated that TSA is able to produce more ordered nanotubes, in comparison to OSA [3]. A common method of two stages of anodic oxidation is inclusive of (1) the one-step anodization for the development of titania nanotubes, (2) following removal of the fabricated titania nanotubes arrays via ultrasonic treatment, and (3) the two-step anodization to fabricate titania nanotubes from the same substrate [4]. However, the remained hemispherical concaves left due the peeling off of anodic titania nanotubes fabricated by one step anodization, are unable to guide the growth of nanopores at the anodizing process of the two-step anodization in some cases. It is noted that the height of arrays formed will not escalate when the rate of oxidation at the titanium oxide link at the bottom is the same as the proportion of disintegration at the oxide/electrolyte interface at the top. Thus, the porous film on the top of the titania nanotubes arrays is responsible as the guarding layer from disintegration, and therefore the formation of lengthen nanotubes is feasible.

2. METHODOLOGY

Samples of 4 cm × 1 cm T foils (99.6% purity, 1 mm thick, Nilaco) were ultrasonically cleaned with acetone, ethanol and deionized (DI) water for 10 min, respectively. First step anodization was carried out in electrolyte solution containing 100 ml ethylene glycol (99.5%, Merck), 0.3 wt% of NH₄F (98%, Sigma Aldrich). The anodization process was carried out using an electrochemical cell consisting of two electrodes where Ti substrate as anode and Carbon electrode as cathode. The first anodization process was using 40 V for 30 minutes at room temperature. The nanotube layer formed was removed by ultrasonication in the Deionized water for 20 min. The removed substrate was used again as anode for the Second-step anodization in the previous solution electrolyte. Then, the formed TiO₂ NTs were cleaned by ethanol and deionized water to remove excess electrolyte before drying it by using air gun. To enhance the crystallinity of TiO₂ NTs, they were annealed at 400 °C for 1 h in air (heating rate: 10 °C/min). Morphology of the TiO₂ NTs was studied using a high-resolution Field Emission Scanning Electron Microscopy (FESEM). X-ray diffraction (XRD).

3. RESULT AND DISCUSSION

The morphology of the TiO₂ nanotubes formed by two-step anodization is shown in Figure 1. Figure 1 (a) shows the surface morphology while Figure 1 (b) is the cross-section morphology. TiO₂ NTs, grown by this process shows a highly ordered hexagonal closely packed structure (Figure 1 (a)). This is due to the textured substrates pattern. Meanwhile the length for this nanotubes is 3.54 μm (Figure 1 (b)). The two-step anodization process was used to improve the structural order of TiO₂ NTs.

To study the structure and properties of the nanotubes formed, XRD analysis was carried out. Figure 2 show the XRD spectra of TiO₂ nanotubes for as anodized and annealed at 400 °C for 1 h. The crystalline behavior of the TiO₂ NTs prepared by one-step and two-step anodization do not show significant differences [5]. for the as anodized samples there is no oxide peak can be found. But for annealed samples, anatase peaks is seen at 2θ=25° and 38° and can be indexed to (1 0 1), (0 0 4).

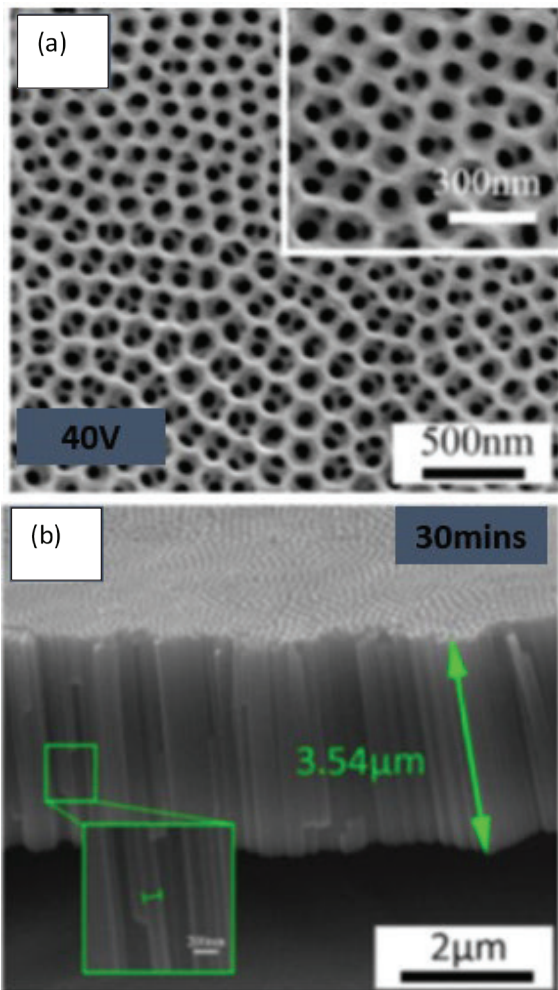


Figure 1 FESEM micrograph of TiO₂ nanotubes anodized at 40V for and 30 minutes (a) top surface (b) cross sectional view

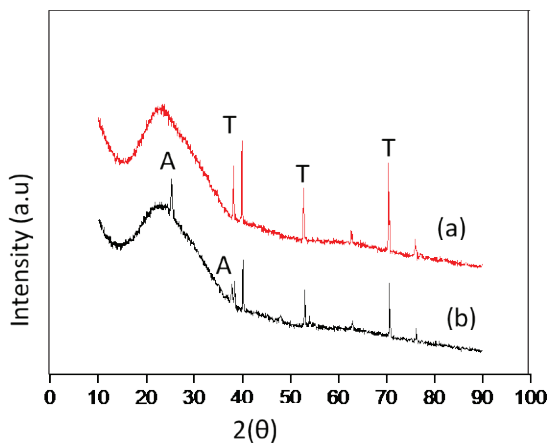


Figure 2 XRD spectra of TiO₂ nanotubes anodized at 40V for and 30 minutes (a) as anodize (b) annealing at 400°C 1 h

physical properties of the obtained TiO₂ NTs was studied.

ACKNOWLEDGEMENT

Authors are grateful to Universiti Teknikal Malaysia Melaka for facilities and technical support.

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4. CONCLUSIONS

In this present work, the highly ordered hexagonal closely packed TiO₂ nanotubes have been successfully fabricated by the two-step anodization method. Effect of the Ti substrates on the surface morphology and